

FUGITIVE DUST TEST REPORT

IN THE VICINITY OF

**U.S.S. LEAD REFINERY, INC.
EAST CHICAGO, INDIANA**

SEPTEMBER 16, 1985

Conducted By:

**EMISSIONS SAMPLING SECTION
AIR POLLUTION CONTROL DIVISION
INDIANA STATE BOARD OF HEALTH**

EPA Region 5 Records Ctr.



308180

Reference #14

INTRODUCTION

On September 16, 1985, an ambient air pollution study was conducted in the vicinity of U.S.S. Lead Refinery, Inc., located at 5300 Kennedy Avenue, East Chicago, Indiana. The purpose of the study was to determine the compliance status of U.S.S. Lead in regard to Regulation 325 IAC 6-4, concerning fugitive dust emissions. A copy of this regulation can be found in Appendix I. Personnel participating in this study were Ed Surla, Vic Windle and Daniel F. Hancock of the Emissions Sampling Section, Air Pollution Control Division, Indiana State Board of Health.

PROCESS INFORMATION

U.S.S. Lead is a secondary lead smelter. The furnace is charged with lead plates reclaimed from used batteries. The furnace was not in operation during the fugitive dust test.

Potential sources of fugitive dust are storage piles of furnace slag, flue dust and battery casings located on plant property. Fugitive dust may also be generated by traffic within the plant boundaries.

SAMPLING PROCEDURES

The test consisted of simultaneous ambient sampling at two locations, one of which was upwind (background site) of the U.S.S. Lead property, and another which was downwind (primary site) of the plant property. The location of the sampling sites for the test can be found on the map included in Appendix II.

The exposed filters were removed from the samplers and transported to the laboratory. The filters were dessicated 24 hours and then weighed to the nearest milligram.

RESULTS

The calculations for the total suspended particulate determination are shown in Appendix III. Kurz flow controllers attached to each sampler maintain a true air flow. The true air flow rate multiplied by the total sampling time equals the total air volume sampled. The weight of the filter catch divided by the total air volume results in the total suspended particulate concentration. A summary of the test results follows:

TEST DATE: September 16, 1985

PRIMARY SITE PARTICULATE CONCENTRATION: 218 micrograms/cubic meter

BACKGROUND SITE PARTICULATE CONCENTRATION: 122 micrograms/cubic meter

CONCENTRATION INCREASE: 96 micrograms/cubic meter

CONCLUSION

The maximum allowable particulate concentration increase as established in the Indiana Air Pollution Control Board Rule 325 IAC 6-4, Section 2(c), is 50 micrograms per cubic meter (primary over background). The results of this study indicate a concentration increase of 96 micrograms per cubic meter. Therefore, this source is not in compliance with Rule 325 IAC 6-4, Section 2(c).

In addition, the filter samples were analyzed for lead concentration using procedures outlined in EPA Method 12. Lead analysis results are summarized in Appendix IV.

APPENDIX I
REGULATION

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TITLE 325 AIR POLLUTION CONTROL BOARD

Rule 4. Fugitive Dust Emissions - Maximum Allowable

325 IAC 6-4-1 Applicability

Authority: IC 13-1-1-4; IC 13-7-5-1

Affected: IC 13-1-1-1; IC 13-1-1-4; IC 13-7-1-1; IC 13-7-5-1;
IC 13-7-7-2

Sec 1. (a) This Rule shall apply to all sources of fugitive dust. For the purposes of this Rule, "fugitive dust" means the generation of particulate matter to the extent that some portion of the material escapes beyond the property line or boundaries of the property, right-of-way, or easement on which the source is located.

325 IAC 6-4-2 Allowable Emissions

Authority: IC 13-1-1-4; IC 13-7-5-1

Affected: IC 13-1-1-1; IC 13-1-1-4; IC 13-7-1-1; IC 13-7-5-1;
IC 13-7-7-2

Sec. 2. (a) A source or sources generating fugitive dust shall be in violation of this Rule if any of the following criteria are violated:

(1) **Maximum Allowable Particles** - A source or combination of sources which cause to exist fugitive dust concentrations greater than 67% in excess of ambient upwind concentrations as determined by the following formula:

$$P = \frac{100 (R-U)}{U}$$

P = Percentage increase

R = Number of particles of fugitive dust measured at downwind receptor site

U = Number of particles of fugitive dust measured at upwind or background site

(2) **Potential Respiratory Damage** - The fugitive dust is comprised of 50% or more respirable dust, then the percent increase of dust concentration in Section 2(a) shall be modified as follows:

$$P_R = (1.5 - N) P$$

Where N = Fraction of fugitive dust that is respirable dust; P_R = allowable percentage increase in dust concentration above background; and P = no value greater than 67%.

Promulgated 8/27/80

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(3) **Ambient Air Concentrations** - The ground-level ambient air concentrations exceed 50 micrograms per cubic meter above background concentrations for a 60-minute period.

(4) **Visible Emissions** - If fugitive dust is visible crossing the boundary or property line of a source and may be refuted by factual data expressed in Section 2(a), 2(b), or 2(c).

325 IAC 6-4-3 Combined Sources

Authority: IC 13-1-1-4; IC 13-7-5-1

Affected: IC 13-1-1-1; IC 13-1-1-4; IC 13-7-1-1; IC 13-7-5-1;
IC 13-7-7-2

Sec. 3. (a) The allowable particles shall refer to the total of all particles leaving the boundaries or crossing the property lines of any source of fugitive dust regardless of whether from a single operation or a number of operations. If the source is determined to be comprised of two or more legally separate persons, each shall be held proportionately responsible on the basis of contributions by each person as determined by microscopic analysis. In such cases, samples shall be taken downwind from the combination of sources and at the fence line of each source.

(b) No source which is contributing to a combined downwind fugitive dust concentration in excess of the limits of this Rule shall be required to reduce emissions if the concentrations at his property line are in compliance unless all contributors are individually in compliance and a combined fugitive dust concentration still exceeds the limits of this Rule. Each source shall then be required to reduce its emissions by like percentages to achieve an acceptable combined downwind concentration.

(c) When all contributors are individually in compliance and no nuisance to the surrounding community is created, the Board may waive the requirement for further reduction in emissions by combined contributors.

325 IAC 6-4-4 Mobile Fugitive Dust Sources

Authority: IC 13-1-1-4; IC 13-7-5-1

Affected: IC 13-1-1-1; IC 13-1-1-4; IC 13-7-1-1; IC 13-7-5-1;
IC 13-7-7-2

Sec. 4. (a) No vehicle shall be driven or moved on any public street, road, alley, highway, or other thoroughfare, unless such vehicle is so constructed as to prevent its contents from dripping, sifting, leaking, or otherwise escaping therefrom so as to create conditions which result in fugitive dust. This section applies only to the cargo any vehicle may be conveying and mud tracked by the vehicle.

325 IAC 6-4-5 Methods of Measurement

Authority: IC 13-1-1-4; IC 13-7-5-1

Affected: IC 13-1-1-1; IC 13-1-1-4; IC 13-7-1-1; IC 13-7-5-1;
IC 13-7-7-2

Sec. 5. (a) **Particle Numbers and Sizes** - Particle quantities and sizes will be measured by manual microscopic analysis of a dustfall sample collected on a sticky slide or by use of commercially available particle counting

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devices which count and classify particles by micron size range, or other methods acceptable to the Board.

(b) Ambient Air Concentrations - Ambient air concentrations shall be measured using the standard Hi Volume Sampling and Analysis Techniques as specified by EPA in the April 30, 1971, Federal Register.

(c) Visible Emissions - Observations by a qualified representative of the Board of visible emissions crossing the property line of the source at or near ground level.

325 IAC 6-4-6 Exemptions

Authority: IC 13-1-1-4; IC 13-7-5-1

Affected: IC 13-1-1-1; IC 13-1-1-4; IC 13-7-1-1; IC 13-7-5-1;
IC 13-7-7-2

Sec. 6. The following conditions will be considered as exceptions to this Rule and therefore not in violation:

(a) Release of steam not in combination with any other gaseous or particulate pollutants unless the condensation from said steam creates a nuisance or hazard in the surrounding community.

(b) Fugitive dust from publicly maintained unpaved thoroughfares where no nuisance or health hazard is created by its usage or where it is demonstrated to the Board that no means are available to finance the necessary road improvements immediately. A reasonable long-range schedule for necessary road improvements must be submitted to support the Board's granting such an exception.

(c) Fugitive dust from construction or demolition where every reasonable precaution has been taken in minimizing fugitive dust emissions.

(d) Fugitive dust generated from agricultural operations providing every reasonable precaution is taken to minimize emissions and providing operations are terminated if a severe health hazard is generated because of prevailing meteorological conditions.

(e) Visible plumes from a stack or chimney which provide adequate dispersion and are in compliance with other applicable Rules.

(f) Fugitive dust from a source caused by adverse meteorological conditions.

325 IAC 6-4-7 Compliance

Authority: IC 13-1-1-4; IC 13-7-5-1

Affected: IC 13-1-1-1; IC 13-1-1-4; IC 13-7-1-1; IC 13-7-5-1;
IC 13-7-7-2

Sec. 7. (a) All sources must comply with this Rule as soon as practicable but no later than July 1, 1974.

APPENDIX II

SAMPLING SITE LOCATIONS

[illegible]

3.2 MI. NE
LAKE CALUMET

UNITED STATES
DEPARTMENT OF THE INTERIOR
GEOLOGICAL SURVEY

Wind Direction ↑

N 8

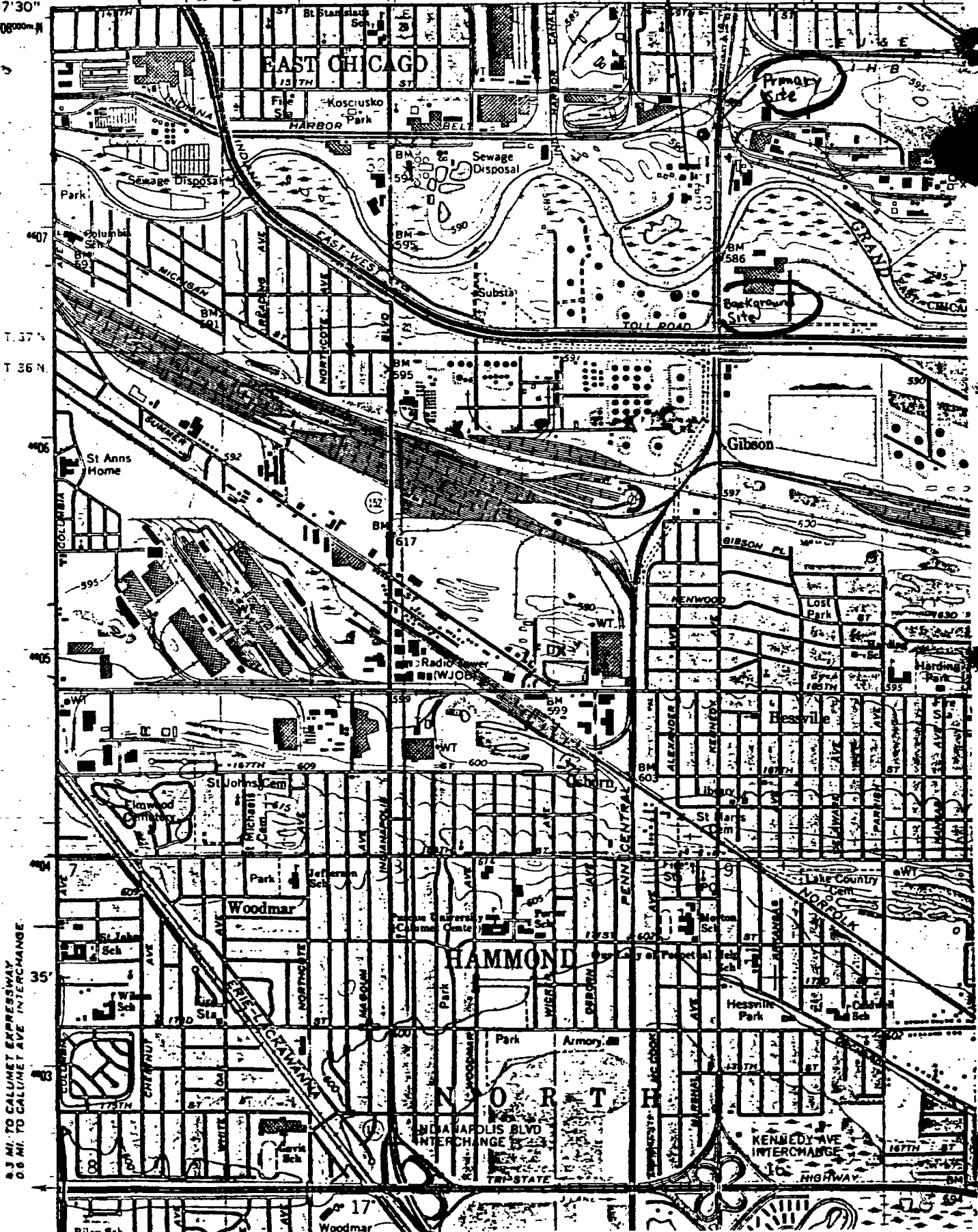
DEF

U.S.S. LEAD CO.

87°30'
41°37'30"

1.3 MI. TO INTERCHANGE 1
CHICAGO, ILL. (LOOP) 23 MI.
WHITING & MI.

27°30' 192



3.3 MI. TO CALUMET EXPRESSWAY
0.6 MI. TO CALUMET AVE INTERCHANGE

HAMMOND

NORTH

KENNEDY AVE INTERCHANGE

16 HIGHWAY

APPENDIX III

CALCULATIONS

TOTAL SUSPENDED PARTICULATE CALCULATIONS

PRIMARY SITE

(1) Observed Air Flow (CFM) - From Flow Meter = _____ CFM

(2) True Air Flow (From Flow Controller) = 1.30 /minute

(3) Weight of Filter Catch (grams) = Final Weight (g) - Initial Weight (g)
 = 4.145 g - 4.111 g
 = 0.034 grams

(4) Adjusted Total Air Volume (cubic meters)
 = True Air Flow (cubic meters/minute) x Time (min.)
 = 1.30 x 120
 = 156 cubic meters

(5) Particulate Concentration (micrograms/cubic meter)
 = $\frac{\text{Weight of Filter Catch (g)}}{\text{Adjusted Total Air Volume (cubic meters)}} \times 10^6$
 = 218 micrograms/cubic meter

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TOTAL SUSPENDED PARTICULATE CALCULATIONS
BACKGROUND SITE

- (1) Observed Air Flow (CFM) - From Flow Meter = _____ CFM
- (2) True Air Flow (Kurz Flow Controller) = 1.30 m³/minute
- (3) Weight of Filter Catch (grams) = Final Weight (g) - Initial Weight (g)
= 4.061 g - 4.042 g
= 0.019 grams
- (4) Adjusted Total Air Volume (cubic meters)
= True Air Flow (cubic meters/minute) x Time (min.)
= 1.30 x 120
= 156 cubic meters
- (5) Particulate Concentration (micrograms/cubic meter)
= $\frac{\text{Weight of Filter Catch (g)}}{\text{Adjusted Total Air Volume (cubic meters)}} \times 10^6$
= 122 micrograms/cubic meter

APPENDIX IV

LEAD ANALYSIS

STATE BOARD OF HEALTH

INDIANAPOLIS

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OFFICE MEMORANDUM

TO: Vic Windle

DATE: October 22, 1985

THRU: L. Fedor
W. Smith
E. Suria

FROM: Phil Zillinger

SUBJECT: Hi-Vol Samples From USS Lead Collected September 16, 1985

The above mentioned samples were analyzed on September 24, 1985 using the procedures outlined in Method 12. The following results were obtained:

<u>Sample</u>	<u>Filter Number</u>	<u>Lead Concentration (ug Pb/m³)</u>
Background	5021364	0.375
Primary	5021359	38.187

PZ/bs

Reference 22

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Federal Register / Vol. 47, No. 74 / Friday, April 16, 1982 / Rules and Regulations

Appendix A—Reference Methods

Method 12. Determination of Inorganic Lead Emissions From Stationary Sources

1. Applicability and Principle.

1.1 Applicability. This method applies to the determination of inorganic lead (Pb) emissions from specified stationary sources only.

1.2 Principle. Particulate and gaseous Pb

2.3 Precision. The within-laboratory precision, as measured by the coefficient of variation ranges from 0.2 to 9.5 percent relative to a run-mean concentration. These values were based on tests conducted at a gray iron foundry, a lead storage battery manufacturing plant, a secondary lead smelter, and a lead recovery furnace of an alkyl lead manufacturing plant. The concentrations encountered during these tests ranged from 0.81 to 123.3 mg Pb/m³.

2.4 Interferences. Sample matrix effects

3.1.1 Probe Nozzle, Probe Liner, Pilot Tube, Differential Pressure Gauge, Filter Holder, Filter Heating System, Metering System, Barometer, and Gas Density Determination Equipment. Same as Method 5, Sections 2.1.1 to 2.1.6 and 2.1.8 to 2.1.10, respectively.

3.1.2 Impingers. Four impingers connected in series with leak-free ground glass fittings or any similar leak-free noncontaminating fittings. For the first, third, and fourth impingers use the Greenburg-Smith design.

Reference 22

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TO

USS Lead Refinery file
Lake Co.

IND047030224

FROM

Dean J. Nygard

SUBJECT

HRS Documentation

MESSAGE

On March 25, 1980, I contacted Mr. Ed Suria of the Division of Air Pollution Control regarding the fugitive dust study at the above referenced facility. Mr. Suria explained that the lead analysis used for this study was U.S. EPA Method 12. I have included this as part of

REPLY

reference 22. Further questions can be addressed to Mr. Ed Suria at AC 317/633-0671.

FOLLOW-UP DATE

081 A W22-SS-3 State Form No. 1352

THE INSTRUMENTS

DRY GAS METER

AIR-TIGHT
PUMP

Figure 12-1. Inorganic lead sampling train.

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RM 12

Revision

4. Reagents

4.1. Reagents

4.1.1 Filter: Gelman Spectro Grade, Reeve Angel 934 AH, MSA 1106 EH, all with lot assay for Pb, or other high-purity glass fiber filters without organic binder, exhibiting at least 99.95 percent efficiency (<0.05 percent

penetration) on 0.3 micron dioctyl phthalate smoke particles. Conduct the filter efficiency test using ASTM Standard Method D2829-71 (incorporated by reference—see § 60.17) or use test data from the supplier's quality control program.

4.1.2

4.1.3 Water: Deionized distilled to conform to ASTM Specification D1231-77 (incorporated by reference—see § 60.17). Type 3. If high concentrations of organic matter are not expected to be present, the analyst may delete the potassium permanganate test for oxidizable organic matter.

3.2 Sample Recovery. The following items are needed:

3.2.1 Probe-Liner and Probe-Nozzle Brushes, Petri Dishes, Plastic Storage Containers, and Funnel and Rubber Policeman. Same as Method 5, Sections 2.2.1, 2.2.4, 2.2.6, and 2.2.7, respectively.

3.2.2 Wash Bottles, Glass (2).

3.2.3 Sample Storage Containers. Chemically resistant, borosilicate glass bottles, for 0.1 nitric acid (HNO_3) impinger and probe solutions and washes, 1000-ml. Use screw-cap liners that are either rubber-backed Teflon® or leak-free and resistant to chemical attack by 0.1 N HNO_3 . (Narrow mouth glass bottles have been found to be less prone to leakage.)

3.2.4 Graduated Cylinder and/or Balance. To measure condensed water to within 2 ml or 1 g. Use a graduated cylinder that has a minimum capacity of 500 ml. and subdivisions no greater than 5 ml. (Most laboratory balances are capable of weighing to the nearest 0.5 g or less.)

3.2.5 Funnel, Glass, to aid in sample recovery.

3.3 Analysis. The following equipment is needed:

3.3.1 Atomic Absorption Spectrophotometer. With lead hollow cathode lamp and burner for air/acetylene flame.

3.3.2 Hot Plate.

3.3.3 Erlenmeyer Flasks, 125-ml, 24/40 S.

3.3.4 Membrane Filters, Millipore SCWPO 4700 or equivalent.

3.3.5 Filtration Apparatus, Millipore vacuum filtration unit, or equivalent, for use with the above membrane filter.

3.3.6 Volumetric Flasks, 100-ml, 250-ml, and 1000 ml.

4. Reagents.

4.1 Sampling. The reagents used in sampling are as follows:

4.1.1 Filter. Gelman Spectro Grade, Reeve Angel 934 AH, MSA 1100 BHL all with lot assay for Pb, or other high-purity glass fiber filters, without organic binder, exhibiting at least 99.95 percent efficiency (<0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles. Conduct the filter efficiency test using ASTM Standard Method D 2980-71 or use test data from the supplier's quality control program.

4.1.2 Silica Gel, Crushed Ice, and Stopcock Grease. Same as Method 5, Section 3.1.2, 3.1.4, and 3.1.5, respectively.

4.1.3 Water. Deionized distilled, to conform to ASTM Specification D 1193-74, Type 3. If high concentrations of organic matter are not expected to be present, the analyst may delete the potassium permanganate test for oxidizable organic matter.

4.1.4 Nitric Acid, 0.1 N. Dilute 0.5 ml of concentrated HNO_3 to 1 liter with deionized distilled water. (It may be desirable to run blanks before field use to eliminate a high blank on test samples.)

4.2 Pretest Preparation. 6 N HNO_3 is needed. Dilute 399 ml of concentrated HNO_3 to 1 liter with deionized distilled water.

4.3 Sample Recovery. 0.1 N HNO_3 (same as 4.1.4 above) is needed for sample recovery.

4.4 Analysis. The following reagents are needed for analysis (use ACS reagent grade chemicals or equivalent, unless otherwise specified):

4.4.1 Water. Same as 4.1.3 above.

4.4.2 Nitric Acid, Concentrated.

4.4.3 Nitric Acid, 50 percent (V/V). Dilute 500 ml of concentrated HNO_3 to 1 liter with deionized distilled water.

4.4.4 Stock Lead Standard Solution, 1000 $\mu\text{g Pb/ml}$. Dissolve 0.1598 g of lead nitrate [$\text{Pb}(\text{NO}_3)_2$] in about 60 ml of deionized distilled water, add 2 ml concentrated HNO_3 , and dilute to 100 ml with deionized distilled water.

4.4.5 Working Lead Standards. Pipet 0.0, 1.0, 2.0, 3.0, 4.0, and 5.0 ml of the stock lead standard solution (4.4.4) into 250-ml volumetric flasks. Add 5 ml of concentrated HNO_3 to each flask and dilute to volume with deionized distilled water. These working standards contain 0.0, 4.0, 8.0, 12.0, 16.0, and 20.0 $\mu\text{g Pb/ml}$, respectively. Prepare, as needed, additional standards at other concentrations in a similar manner.

4.4.6 Air. Suitable quality for atomic absorption analysis.

4.4.7 Acetylene. Suitable quality for atomic absorption analysis.

4.4.8 Hydrogen Peroxide, 3 percent (V/V). Dilute 10 ml of 30 percent H_2O_2 to 100 ml with deionized distilled water.

5. Procedure.

5.1 Sampling. The complexity of this method is such that, in order to obtain reliable results, testers should be trained and experienced with the test procedures.

5.1.1 Pretest Preparation. Follow the same general procedure given in Method 5, Section 4.1.1, except the filter need not be weighed.

5.1.2 Preliminary Determinations. Follow the same general procedure given in Method 5, Section 4.1.2.

5.1.3 Preparation of Collection Train. Follow the same general procedure given in Method 5, Section 4.1.3, except place 100 ml of 0.1 HNO_3 in each of the first two impingers, leave the third impinger empty, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the fourth impinger. Set up the train as shown in Figure 12-1.

5.1.4 Leak-Check Procedures. Follow the general leak-check procedures given in Method 5, Sections 4.1.4.1 (Pretest Leak-Check), 4.1.4.2 (Leak-Checks During the Sample Run), and 4.1.4.3 (Post-Test Leak-Check).

5.1.5 Sampling Train Operation. Follow the same general procedure given in Method 5, Section 4.1.5. For each run, record the data required on a data sheet such as the one shown in EPA Method 5, Figure 5-2.

5.1.6 Calculation of Percent Recovery. Same as Method 5, Section 4.1.6.

5.2 Sample Recovery. Begin proper cleanup procedure as soon as the probe is removed from the stack at the end of the sampling period.

Allow the probe to cool. When it can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it. Do not cap off the probe too tightly while the sampling train

is cooling down as this would create a vacuum in the filter holder, thus drawing liquid from the impingers into the filter.

Before moving the sampling train to the cleanup site, remove the probe from the sampling train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Wipe off the silicone grease from the glassware inlet where the probe was fastened and cap the inlet. Remove the umbilical cord from the last impinger and cap the impinger. The tester may use ground-glass stoppers, plastic caps, or serum caps to close these openings.

Transfer the probe and filter-impinger assembly to a cleanup area, which is clean and protected from the wind so that the chances of contaminating or losing the sample are minimized.

Inspect the train prior to and during disassembly and note any abnormal conditions. Treat the samples as follows:

5.2.1 Container No. 1 (Filter). Carefully remove the filter from the filter holder and place it in its identified petri dish container. If it is necessary to fold the filter, do so such that the sample-exposed side is inside the fold. Carefully transfer to the petri dish any visible sample matter and/or filter fibers that adhere to the filter holder gasket by using a dry Nylon bristle brush and/or a sharp-edged blade. Seal the container.

5.2.2 Container No. 2 (Probe). Taking care that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover sample matter or any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components with 0.1 N HNO_3 , and placing the wash into a glass sample storage container. Measure and record (to the nearest 2-ml) the total amount of 0.1 N HNO_3 used for each rinse. Perform the 0.1 N HNO_3 rinses as follows:

Carefully remove the probe nozzle and rinse the inside surfaces with 0.1 N HNO_3 from a wash bottle while brushing with a stainless steel, Nylon-bristle brush. Brush until the 0.1 N HNO_3 rinse shows no visible particles, then make a final rinse of the inside surface.

Brush and rinse with 0.1 N HNO_3 the outside parts of the Swagelok fitting in a similar way until no visible particles remain.

Rinse the probe liner with 0.1 N HNO_3 . While rotating the probe so that all inside surfaces will be rinsed with 0.1 N HNO_3 , tilt the probe and squirt 0.1 N HNO_3 into its upper end. Let the 0.1 N HNO_3 drain from the lower end into the sample container. The tester may use a glass funnel to aid in transferring liquid washes to the container. Follow the rinse with a probe brush. Hold the probe in an inclined position, squirt 0.1 N HNO_3 into the upper end of the probe as the probe brush is being pushed with a twisting action through the probe, hold the sample container underneath the lower end of the probe and catch any 0.1 N HNO_3 and sample matter that is brushed from the probe. Run the brush through the probe three times or more until no visible sample matter is carried out with the 0.1 N HNO_3 , and make routine on the probe liner on visual inspection. Wash...

*Mention of trade names or specific products does not constitute endorsement by the U.S. Environmental Protection Agency.

stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times, since metal probes have small crevices in which sample matter can be entrapped. Rinse the brush with 0.1 N HNO₃ and quantitatively collect these washings in the sample container. After the brushing make a final rinse of the probe as described above.

It is recommended that two people clean the probe to minimize loss of sample. Between sampling runs, keep brushes clean and protected from contamination.

After insuring that all joints are wiped clean of silicone grease, brush and rinse with 0.1 N HNO₃ the inside of the front half of the filter holder. Brush and rinse each surface three times or more, if needed, to remove visible sample matter. Make a final rinse of the brush and filter holder. After all 0.1 N HNO₃ washings and sample matter are collected in the sample container, tighten the lid on the sample container so that the fluid will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether leakage occurs during transport. Label the container to clearly identify its contents.

5.2.3 Container No. 3 (Silica Gel). Check the color of the indicating silica gel to determine if it has been completely spent and make a notation of its condition. Transfer the silica gel from the fourth impinger to the original container and seal. The tester may use a funnel to pour the silica gel and a rubber policeman to remove the silica gel from the impinger. It is not necessary to remove the small amount of particles that may adhere to the walls and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, the tester may follow procedure for Container No. 3 under Section 5.4 (Analysis).

5.2.4 Container No. 4 (Impingers). Due to the large quantity of liquid involved, the tester may place the impinger solutions in several containers. Clean each of the first three impingers and connecting glassware in the following manner:

1. Wipe the impinger ball joints free of silicone grease and cap the joints.
2. Rotate and agitate each impinger, so that the impinger contents might serve as a rinse solution.
3. Transfer the contents of the impingers to a 500-ml graduated cylinder. Remove the outlet ball joint cap and drain the contents through this opening. Do not separate the impinger parts (inner and outer tubes) while transferring their contents to the cylinder. Measure the liquid volume to within ± 2 ml. Alternatively, determine the weight of the liquid to within ± 0.5 g. Record in the log the volume or weight of the liquid present, along with a notation of any color or film observed in the impinger catch. The liquid volume or weight is needed, along with the silica gel data, to calculate the stack gas moisture content (see Method 5, Figure 5-3).
4. Transfer the contents to Container No. 4.
5. Note: In steps 3 and 4 below, measure and record the total amount of 0.1 N HNO₃ used for rinsing. Pour approximately 30 ml of 0.1 N HNO₃ into each of the first three

impingers and agitate the impingers. Drain the 0.1 N HNO₃ through the outlet arm of each impinger into Container No. 4. Repeat this operation a second time; inspect the impingers for any abnormal conditions.

6. Wipe the ball joints of the glassware connecting the impingers free of silicone grease and rinse each piece of glassware twice with 0.1 N HNO₃; transfer this rinse into Container No. 4. (Do not rinse or brush the glass-fritted filter support.) Mark the height of the fluid level to determine whether leakage occurs during transport. Label the container to clearly identify its contents.

5.2.5 Blanks. Save 200 ml of the 0.1 N HNO₃ used for sampling and cleanup as a blank. Take the solution directly from the bottle being used and place into a glass sample container labeled "0.1 N HNO₃, blank."

5.3 Sample Preparation.

5.3.1 Container No. 1 (Filter). Cut the filter into strips and transfer the strips and all loose particulate matter into a 125-ml Erlenmeyer flask. Rinse the petri dish with 10 ml of 50 percent HNO₃ to insure a quantitative transfer and add to the flask. (Note: If the total volume required in Section 5.3.3 is expected to exceed 80 ml, use a 250-ml Erlenmeyer flask in place of the 125-ml flask.)

5.3.2 Containers No. 2 and No. 4 (Probe and Impingers). (Check the liquid level in Containers No. 2 and/or No. 4 and confirm as to whether or not leakage occurred during transport; note observation on the analysis sheet. If a noticeable amount of leakage had occurred, either void the sample or take steps, subject to the approval of the Administrator, to adjust the final results.) Combine the contents of Containers No. 2 and No. 4 and take to dryness on a hot plate.

5.3.3 Sample Extraction for lead. Based on the approximate stack gas particulate concentration and the total volume of stack gas sampled, estimate the total weight of particulate sample collected. Then transfer the residue from Containers No. 2 and No. 4 to the 125-ml Erlenmeyer flask that contains the filter using rubber policeman and 10 ml of 50 percent HNO₃ for every 100 mg of sample collected in the train or a minimum of 20 ml of 50 percent HNO₃, whichever is larger.

Place the Erlenmeyer flask on a hot plate and heat with periodic stirring for 30 min at a temperature just below boiling. If the sample volume falls below 15 ml, add more 50 percent HNO₃. Add 10 ml of 3 percent H₂O₂ and continue heating for 10 min. Add 50 ml of hot (80°C) deionized distilled water and heat for 20 min. Remove the flask from the hot plate and allow to cool. Filter the sample through a Millipore membrane filter or equivalent and transfer the filtrate to a 250-ml volumetric flask. Dilute to volume with deionized distilled water.

5.3.4 Filter Blank. Determine a filter blank using two filters from each lot of filters used in the sampling train. Cut each filter into strips and place each filter in a separate 125-ml Erlenmeyer flask. Add 15 ml of 50 percent HNO₃ and treat as described in Section 5.3.3 using 10 ml of 3 percent H₂O₂ and 50 ml of hot deionized distilled water. Filter and dilute to a total volume of 100 ml using deionized distilled water.

5.3.5 0.1 N HNO₃, Blank. Take the entire 200 ml of 0.1 N HNO₃ to dryness on a steam

bath, add 15 ml of 50 percent HNO₃ and treat as described in Section 5.3.3 using 10 ml of 3 percent H₂O₂ and 50 ml of hot, deionized distilled water. Dilute to a total volume of 100 ml using deionized distilled water.

5.4 Analysis.

5.4.1 Lead Determination. Calibrate the spectrophotometer as described in Section 6.2 and determine the absorbance for each source sample, the filter blank, and 0.1 N HNO₃, blank. Analyze each sample three times in this manner. Make appropriate dilutions, as required, to bring all sample Pb concentrations into the linear absorbance range of the spectrophotometer.

If the Pb concentration of a sample is at the low end of the calibration curve and high accuracy is required, the sample can be taken to dryness on a hot plate and the residue dissolved in the appropriate volume of water to bring it into the optimum range of the calibration curve.

5.4.2 Mandatory Check for Matrix Effects on the Lead Results. The analysis for Pb by atomic absorption is sensitive to the chemical composition and to the physical properties (viscosity, pH) of the sample (matrix effects). Since the Pb procedure described here will be applied to many different sources, many sample matrices will be encountered. Thus, check (mandatory) at least one sample from each source using the Method of Additions to ascertain that the chemical composition and physical properties of the sample did not cause erroneous analytical results.

Three acceptable "Method of Additions" procedures are described in the General Procedure Section of the Perkin Elmer Corporation Manual (see Citation 9.1). If the results of the Method of Additions procedure on the source sample do not agree within 5 percent of the value obtained by the conventional atomic absorption analysis, then the tester must reanalyze all samples from the source using the Method of Additions procedure.

5.4.3 Container No. 3 (Silica Gel). The tester may conduct this step in the field. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g; record this weight.

6. Calibration.

Maintain a laboratory log of all calibrations.

6.1 Sampling Train Calibration. Calibrate the sampling train components according to the indicated sections of Method 5: Probe Nozzle (Section 5.1); Pitot Tube (Section 5.2); Metering System (Section 5.3); Probe Heater (Section 5.4); Temperature Gauges (Section 5.5); Leak-Check of the Metering System (Section 5.6); and Barometer (Section 5.7).

6.2 Spectrophotometer. Measure the absorbance of the standard solutions using the instrument settings recommended by the spectrophotometer manufacturer. Repeat until good agreement (± 3 percent) is obtained between two consecutive readings. Plot the absorbance (y-axis) versus concentration in $\mu\text{g Pb/ml}$ (x-axis). Draw or compute a straight line through the linear portion of the curve. Do not force the calibration curve through zero, but if the curve does not pass through the origin, or at least be closer to the origin than ± 0.005

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torbance units, check for incorrectly prepared standards and for curvature in the calibration curve.

To determine stability of the calibration curve, run a blank and a standard after every five samples and recalibrate, as necessary.

7. Calculations.

7.1 Dry Gas Volume. Using the data from this test, calculate $V_{\text{m(tot)}}$, the total volume of dry gas metered corrected to standard conditions (20°C and 760 mm Hg), by using Equation 5-1 of Method 5. If necessary, adjust $V_{\text{m(tot)}}$ for leakages as outlined in Section 6.3 of Method 5. See the field data sheet for the average dry gas meter temperature and average orifice pressure drop.

7.2 Volume of Water Vapor and Moisture Content. Using data obtained in this test and Equations 5-2 and 5-3 of Method 5, calculate the volume of water vapor $V_{\text{w(tot)}}$ and the moisture content B_w of the stack gas.

7.3 Total Lead in Source Sample. For each source sample correct the average absorbance for the contribution of the filter blank and the 0.1 N HNO₃ blank. Use the calibration curve and this corrected absorbance to determine the $\mu\text{g Pb}$ concentration in the sample aspirated into the spectrophotometer. Calculate the total Pb content C_{Pb} (in μg) in the original source sample; correct for all the dilutions that were made to bring the Pb concentration of the sample into the linear range of the spectrophotometer.

7.4 Lead Concentration. Calculate the stack gas Pb concentration C_{Pb} in mg/dscm as follows:

$$C_{\text{Pb}} = K \frac{C_{\text{Pb}}}{V_{\text{m(tot)}}}$$

Where:

$K = 0.001 \text{ mg}/\mu\text{g}$ for metric units.

$= 2.205 \text{ lb}/\mu\text{g}$ for English units.

7.5 Isokinetic Variation and Acceptable Results. Same as Method 5, Sections 6.11 and 6.12, respectively. To calculate v_a , the average stack gas velocity, use Equation 2-9 of Method 2 and the data from this field test.

8. Alternative Test Methods for Inorganic Lead.

8.1 Simultaneous Determination of Particulate and Lead Emissions. The tester may use Method 5 to simultaneously determine Pb provided that (1) he uses acetone to remove particulate from the probe and inside of the filter holder as specified by Method 5, (2) he uses 0.1 N HNO₃ in the impingers, (3) he uses a glass fiber filter with a low Pb background, and (4) he treats and analyzes the entire train contents, including the impingers, for Pb as described in Section 5 of this method.

8.2 Filter Location. The tester may use a filter between the third and fourth impinger provided that he includes the filter in the analysis for Pb.

8.3 In-stack Filter. The tester may use an in-stack filter; provided that (1) he uses a

glass-lined probe and at least two impingers, each containing 100 ml of 0.1 N HNO₃, after the in-stack filter and (2) he recovers and analyzes the probe and impinger contents for Pb. Recover sample from the nozzle with acetone if a particulate analysis is to be made.

9. Bibliography

9.1 Perkin Elmer Corporation. Analytical Methods for Atomic Absorption Spectrophotometry. Norwalk, Connecticut. September 1976.

9.2 American Society for Testing and Materials. Annual Book of ASTM Standards. Part 31; Water, Atmospheric Analysis, Philadelphia, Pa. 1974. p. 40-42.

9.3 Klein, R. and C. Huch. Standard Additions—Uses and Limitations in Spectrophotometric Analysis. *Amer. Lab.* 9:21-27, 1977.

9.4 Mitchell, W.J. and M.R. Midgett. Determining Inorganic and Alkyl Lead Emissions from Stationary Sources. U.S. Environmental Protection Agency. Emission Monitoring and Support Laboratory. Research Triangle Park, N.C. (Presented at National APCA Meeting, Houston, June 20, 1974).

9.5 Same as Method 5, Citations 2 to 5 and 7 of Section 7.

(Secs. 111, 114, and 301(a) of the Clean Air Act as amended (42 U.S.C. 7411, 7414, and 7601(a)))

(FR Doc. 83-10461 Filed 4-13-83; 8:43 am)

ENVIRONMENTAL PROTECTION AGENCY

40 CFR Part 60

[AD-FRL-2620-3]

Standards of Performance for New Stationary Sources; Reference Methods; Revision to Method 12 for Inorganic Lead To Add a Method of Additions Procedure

AGENCY: Environmental Protection Agency (EPA).

ACTION: Final rule.

SUMMARY: The purpose of this action is to promulgate a revision to Method 12 for inorganic lead of Appendix A of 40 CFR Part 60 to include a method of additions procedure, which deals with the resolution of any possible interferences in the lead analysis. This revision is necessary because it has been determined that the method of additions procedures previously cited by Method 12 may not be readily available to the analyst, and were not suitable for incorporation by reference. This revision was proposed in the Federal Register on December 12, 1983 (48 FR 55395). No changes in the revision have been made since proposal, as no comment letters were received.

EFFECTIVE DATE: August 24, 1984.

Under section 307(b)(1) of the Clean Air Act, judicial review of this revision is available *only* by the filing of a petition for review in the U.S. Court of Appeals for the District of Columbia Circuit within 60 days of today's publication of this rule. Under section 307(b)(2) of the Clean Air Act, the requirements that are the subject of today's notice may not be challenged later in civil or criminal proceedings brought by EPA to enforce these requirements.

ADDRESSES: Docket. A docket, number A-83-38, containing materials considered by EPA in development of the promulgated rulemaking, is available for public inspection between 8:00 a.m. and 4:00 p.m., Monday through Friday, at EPA's Central Docket Section (LE-131), West Tower Lobby, Gallery 1, 401 M Street, SW., Washington, D.C. 20460. A reasonable fee may be charged for copying.

FOR FURTHER INFORMATION CONTACT: William Grimley or Roger Shigehara, Emission Measurement Branch, Emission Standards and Engineering Division (MD-19), U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711, telephone (919) 541-2237.

SUPPLEMENTARY INFORMATION: This rulemaking does not impose any additional emission measurement requirements on facilities affected by this rulemaking. Rather, this rulemaking adds a supplementary analytical procedure to a test method that would apply irrespective of this rulemaking. This addition is necessary because the supplementary analytical procedure, which was previously cited by the method, is not suitable for incorporation by reference.

Public Participation

The revision was proposed and published in the Federal Register on December 12, 1983 (48 FR 55395). The opportunity to request a public hearing was presented to provide interested persons the opportunity for oral presentation of data, views, or arguments concerning the proposed revision, but no person desired to make an oral presentation. The public comment period was from December 12, 1983, to February 27, 1984. No comment letters were received.

Docket

The docket is an organized and complete file of the information considered by EPA in the development of this rulemaking. The docket is a dynamic file, since material is added throughout the rulemaking development. The docketing system is intended to allow members of the public and industries involved to identify readily and locate documents so that they can intelligently and effectively participate in the rulemaking process. Along with the statement of basis and purpose of the proposed and promulgated rule and EPA responses to significant comments, the contents of the docket will serve as the record in case of judicial review (Section 307(d)(7)(A)).

Miscellaneous

Under Executive Order 12291, EPA must judge whether a regulation is "major" and therefore subject to the requirement of a regulatory impact analysis. This regulation is not major because it will not have an annual effect on the economy of \$100 million or more; it will not result in a major increase in costs or prices; and there will be no significant adverse effects on competition, employment, investment, productivity innovation, or on the ability of U.S.-based enterprises to compete with foreign-based enterprises in domestic or export markets. This regulation was submitted to OMB for review under E.O. 12291.

Pursuant to the provisions of 5 U.S.C. 605(u), I hereby certify that the attached

rule will not have a significant economic impact on small entities because no additional costs will be incurred. This rule does not contain any information collection requirements subject to OMB review under the Paperwork Reduction Act of 1980 U.S.C. 3501 *et seq.*

This rulemaking is issued under the authority of sections 111, 114, and 301(a) of the Clean Air Act, as amended (42 U.S.C. 7411, 7414, and 7601(a)).

List of Subjects in 40 CFR Part 60

Air pollution control, Aluminum, Ammonium sulfate plants, Asphalt, Cement industry, Coal Copper, Electric power plants, Glass and glass products, Grains, Intergovernmental relations, Iron, Lead, Metals, Metallic Minerals, Motor vehicles, Nitric acid plants, Paper and paper products industry, Petroleum, Phosphate, Sewage disposal, Steel Sulfuric acid plants, Waste treatment and disposal, Zinc, Tires, Incorporation by Reference, Can surface coating, Sulfuric acid plants, Industrial organic chemicals, Organic solvent cleaners, Fossil fuel-fired steam generators, Fiberglass Insulation, Synthetic Fibers, Lime.

Dated: August 2, 1984.

Alvin L. Alm,

Acting Administrator.

PART 60—[AMENDED]

40 CFR Part 60, Appendix A, Method 12, is amended as follows:

1. By revising Section 5.4.2 to read as follows:

5.4.2 Check for Matrix Effects on the Lead Results.

Since the analysis for Pb by atomic absorption is sensitive to the chemical composition and to the physical properties (viscosity, pH) of the sample (matrix effects), the analyst shall check at least one sample from each source using the method of additions as follows:

Add or spike an equal volume of standard solution to an aliquot of the sample solution, then measure the absorbance of the resulting solution and the absorbance of an aliquot of unspiked sample.

Next, calculate the Pb concentration C_s in $\mu\text{g/ml}$ of the sample solution by using the following equation:

$$C_s = C_a \frac{A_s}{A_s - A_u} \quad \text{Eq. 12-1}$$

Where:

C_s = Pb concentration of the standard solution, $\mu\text{g/ml}$.

rate tables, which are proposed amendments to the International Mail Manual (incorporated by reference in the Code of Federal Regulations, 39 CFR 10.1), and which are to become effective on the date service begins. No comments were received.

Accordingly, the Postal Service states that it intends to begin Express Mail International Service with Norway on October 23, 1984 at the rates indicated in the table below.

List of Subjects in 39 CFR Part 10

Postal service, Foreign relations.

NORWAY.—EXPRESS MAIL INTERNATIONAL SERVICE

Custom designed service ^{1,2}		On demand service ³	
Up to and including		Up to and including	
Pounds	Rate	Pounds	Rate
1	\$28.00	1	\$20.00
2	31.70	2	23.70
3	35.40	3	27.40
4	39.10	4	31.10
5	42.80	5	34.80
6	46.50	6	38.50
7	50.20	7	42.20
8	53.90	8	45.90
9	57.60	9	49.60
10	61.30	10	53.30
11	65.00	11	57.00
12	68.70	12	60.70
13	72.40	13	64.40
14	76.10	14	68.10
15	79.80	15	71.80
16	83.50	16	75.50
17	87.20	17	79.20
18	90.90	18	82.90
19	94.60	19	86.60
20	98.30	20	90.30
21	102.00	21	94.00
22	105.70	22	97.70
23	109.40	23	101.40
24	113.10	24	105.10
25	116.80	25	108.80
26	120.50	26	112.50
27	124.20	27	116.20
28	127.90	28	119.90
29	131.60	29	123.60
30	135.30	30	127.30
31	139.00	31	131.00
32	142.70	32	134.70
33	146.40	33	138.40
34	150.10	34	142.10
35	153.80	35	145.80
36	157.50	36	149.50
37	161.20	37	153.20
38	164.90	38	156.90
39	168.60	39	160.60
40	172.30	40	164.30
41	176.00	41	168.00
42	179.70	42	171.70
43	183.40	43	175.40
44	187.10	44	179.10

¹ Rates in this table are applicable to each piece of International Custom Designed Express Mail shipped under a Service Agreement providing for tender by the customer at a designated Post Office.

² Pickup is available under a Service Agreement for an add-on charge of \$5.00 for each pickup stop, regardless of the number of pieces picked up. Domestic and International Express Mail picked up together under the same Service Agreement incurs only one pickup charge.

A transmittal letter making these changes in the pages of the International Mail Manual will be published in the Federal Register as provided in 39 CFR 10.3 and will be transmitted to subscribers automatically.

(39 U.S.C. 401, 404, 407)

W. Allen Sanders,

Associate General Counsel, Office of General Law and Administration.

[FR Doc. 84-23247 Filed 9-21-84; 8:45 am]

BILLING CODE 7710-12-M

ENVIRONMENTAL PROTECTION AGENCY

40 CFR Part 60

[AD-FRL-2620-31]

Standards of Performance for New Stationary Sources; Reference Methods; Revision To Method 12 for Inorganic Lead To Add a Method of Additions Procedure

Correction

In FR Doc. 84-21131 beginning on page 33842 in the issue of Friday, August 24, 1984, make the following correction: In column three, the equation at the bottom of the page should read:

$$C_s = C_a \frac{A_s}{A_c - A_s}$$

BILLING CODE 1905-91-M

DEPARTMENT OF TRANSPORTATION

Coast Guard

46 CFR Part 170

[CGD 79-023]

Subdivision and Stability Regulations

AGENCY: Coast Guard, DOT.

ACTION: Final rule; correction.

SUMMARY: This document corrects three incorrect definitions in the final rule issued November 4, 1983.

FOR FURTHER INFORMATION CONTACT: Lt. Albert W. Horsmon Jr., Commandant (G-MTH-S/13), Room 1308, U.S. Coast Guard Headquarters, Washington, D.C. 20593, (202) 426-2187.

Discussion of Correction

§ 170.170 (Amended)

In the Federal Register of November 4, 1983, page 51014, some definitions for the coefficient P in the inequality defining GM in § 170.170(a) are incorrect due to typographical errors. The second, third, and fourth definitions for P are corrected to read:

$P = .055 + (L/1309)^2$ metric tons/m³ . . . for ocean service, Great Lakes winter service, or service on exposed waters.

$P = .0033 + (L/14,200)^2$ tons/ft³ . . . for Great Lakes summer service or service on partially protected waters.

$P = .036 + (L/1309)^2$ metric tons/m³ . . . for Great Lakes summer service or service on partially protected waters.

Dated: September 18, 1984.

Clyde T. Lusk, Jr.,

Rear Admiral, U.S. Coast Guard, Chief, Office of Merchant Marine Safety.

[FR Doc. 84-23212 Filed 9-21-84; 8:45 am]

BILLING CODE 4910-14-M

FEDERAL COMMUNICATIONS COMMISSION

47 CFR Part 1

Amendment of Part 1 of the Rules Concerning Practice and Procedure in the Private Radio Services

AGENCY: Federal Communications Commission.

ACTION: Final rule; correction.

SUMMARY: This document corrects an error in the Appendix to an amendment of the rules of practice and procedure in the Private Radio Services.

FOR FURTHER INFORMATION CONTACT: Robert DeYoung, Private Radio Bureau, (202) 632-7175.

Mary Beth Hess, Private Radio Bureau, (202) 634-2443.

SUPPLEMENTARY INFORMATION

Erratum

Released: September 17, 1984.

In the matter of amendment of Part 1 of the Rules concerning Practice and Procedure in the Private Radio Services.

On July 24, 1984, the Commission released an Order (FCC 84-323) (August 2, 1984, 49 FR 30943) in the above-captioned proceeding. Section 1.925(f), (g) and (h) were printed incorrectly in the Appendix. Those paragraphs should read:

§ 1.925 Application for special temporary authorization, temporary permit or temporary operating authority.

(f) An applicant for a Ship Radio station license may operate the radio station pending issuance of the ship station authorization by the Commission for a period of 90 days, under a temporary operating authority, evidenced by a properly executed certification made on FCC Form 506-A.

(g) An applicant for a Business Radio station license utilizing an already authorized facility may operate the radio station for a period of 180 days, under a temporary permit, evidenced by

Appendix A—Reference Methods

Method 12. Determination of Inorganic Lead Emissions From Stationary Sources

1. Applicability and Principle.

1.1 Applicability. This method applies to the determination of inorganic lead (Pb) emissions from specified stationary sources only.

1.2 Principle. Particulate and gaseous Pb emissions are withdrawn isokinetically from the source and collected on a filter and in nitro nitric acid. The collected samples are digested in acid solution and analyzed by atomic absorption spectrometry using an air acetylene flame.

2. Range, Sensitivity, Precision, and Interferences.

2.1 Range. For a minimum analytical accuracy of ± 10 percent, the lower limit of a range is 100 μg . The upper limit can be considerably extended by dilution.

2.2 Analytical Sensitivity. Typical sensitivities for a 1-percent change in absorption (0.0044 absorbance units) are 0.2 and 0.5 $\mu\text{g Pb/ml}$ for the 217.0 and 283.3 nm, respectively.

2.3 Precision. The within-laboratory precision, as measured by the coefficient of variation ranges from 0.2 to 9.5 percent relative to a run-mean concentration. These values were based on tests conducted at a gray iron foundry, a lead storage battery manufacturing plant, a secondary lead smelter, and a lead recovery furnace of an alkyl lead manufacturing plant. The concentrations encountered during these tests ranged from 0.61 to 123.3 mg Pb/m³.

2.4 Interferences. Sample matrix effects may interfere with the analysis for Pb by flame atomic absorption. If this interference is suspected, the analyst may confirm the presence of these matrix effects and frequently eliminate the interference by using the Method of Standard Additions.

High concentrations of copper may interfere with the analysis of Pb at 217.0 nm. This interference can be avoided by analyzing the samples at 283.3 nm.

3. Apparatus.

3.1 Sampling Train. A schematic of the sampling train is shown in Figure 12-1; it is similar to the Method 5 train. The sampling train consists of the following components:

3.1.1 Probe Nozzle, Probe Liner, Pilot Tube, Differential Pressure Gauge, Filter Holder, Filter Heating System, Metering System, Barometer, and Gas Density Determination Equipment. Same as Method 5, Sections 2.1.1 to 2.1.6 and 2.1.8 to 2.1.10, respectively.

3.1.2 Impingers. Four impingers connected in series with leak-free ground glass fittings or any similar leak-free noncontaminating fittings. For the first, third, and fourth impingers, use the Greenburg-Smith design, modified by replacing the tip with a 1.3 cm (1/2 in.) ID glass tube extending to about 1.3 cm (1/2 in.) from the bottom of the flask. For the second impinger, use the Greenburg-Smith design with the standard tip. Place a thermometer, capable of measuring temperature to within 1°C (2°F) at the outlet of the fourth impinger for monitoring purposes.

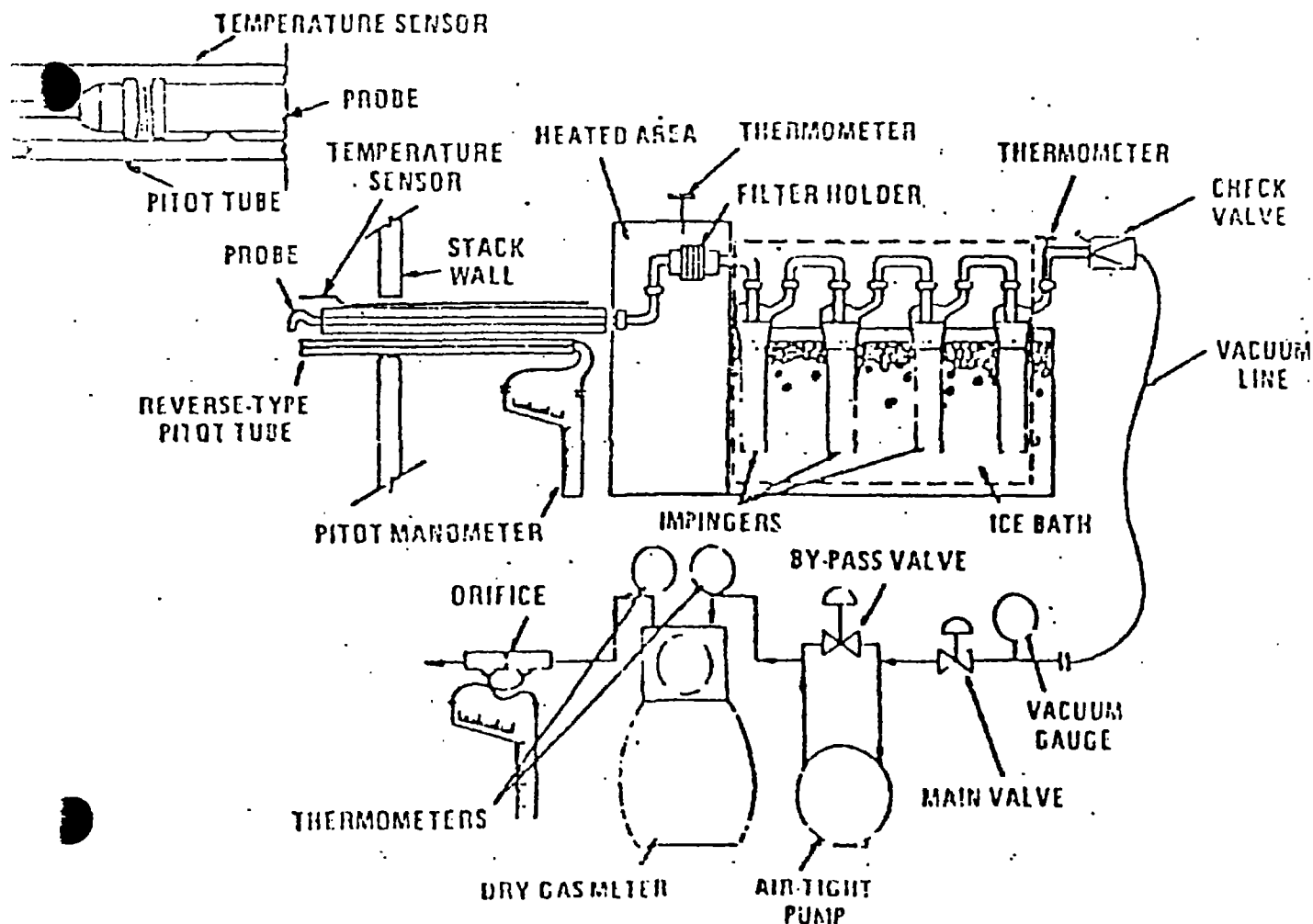


Figure 12-1. Inorganic lead sampling train.

RM 12

Revision

4. Reagents

4.1

4.1.1 Filter. Gelman Spectro Grade, Reeve Angel 934 AH, MSA 1106 EH, all with lot assay for Pb, or other high-purity glass fiber filters without organic binder, exhibiting at least 99.95 percent efficiency (<0.05 percent

penetration) on 0.3 micron dioctyl phthalate smoke particles. Conduct the filter efficiency test using ASTM Standard Method D2639-71 (incorporated by reference—see § 60.17) or the test data from the supplier's quality control program.

4.1.2

4.1.3 Water. Deionized distilled, to conform to ASTM Specification D1591-77 (incorporated by reference—see § 60.17). Type 3. If high concentrations of organic matter are not expected to be present, the analyst may delete the potassium permanganate test for oxidizable organic matter.

i-27-83

3.2 Sample Recovery: The following items are needed:

3.2.1 Probe-Liner and Probe-Nozzle Brushes, Petri Dishes, Plastic Storage Containers, and Funnel and Rubber Policeman. Same as Method 5, Sections 2.2.1, 2.2.4, 2.2.6, and 2.2.7, respectively.

3.2.2 Wash Bottles, Glass (2).

3.2.3 Sample Storage Containers. Chemically resistant, borosilicate glass bottles, for 0.1 nitric acid (HNO_3) impinger and probe solutions and washes, 1000-ml. Use screw-cap liners that are either rubber-backed Teflon® or leak-free and resistant to chemical attack by 0.1 N HNO_3 . (Narrow mouth glass bottles have been found to be less prone to leakage.)

3.2.4 Graduated Cylinder and/or Balance. To measure condensed water to within 2 ml or 1 g. Use a graduated cylinder that has a minimum capacity of 500 ml and subdivisions no greater than 5-ml. (Most laboratory balances are capable of weighing to the nearest 0.5 g or less.)

3.2.5 Funnel, Glass, to aid in sample recovery.

3.3 Analysis: The following equipment is needed:

3.3.1 Atomic Absorption Spectrophotometer, with lead hollow cathode lamp and burner for air/acetylene flame.

3.3.2 Hot Plate.

3.3.3 Erlenmeyer Flasks, 125-ml, 24/40 S.

3.3.4 Membrane Filters, Millipore SCWPO 4700 or equivalent.

3.3.5 Filtration Apparatus, Millipore vacuum filtration unit, or equivalent, for use with the above membrane filter.

3.3.6 Volumetric Flasks, 100-ml, 250-ml, and 1000 ml.

4. Reagents:

4.1 Sampling: The reagents used in sampling are as follows:

4.1.1 Filter: Gelman Spectro Grade, Reeve Angel 934 AH, MSA 1100 BH, all with lot assay for Pb, or other high-purity glass fiber filters, without organic binder, exhibiting at least 99.95 percent efficiency (<0.05 percent penetration) on 0.3 micron diocetyl phthalate smoke particles. Conduct the filter efficiency test using ASTM Standard Method D 2980-71 or use test data from the supplier's quality control program.

4.1.2 Silica Gel, Crushed Ice, and Stopcock Grease. Same as Method 5, Section 3.1.2, 3.1.4, and 3.1.5, respectively.

4.1.3 Water, Deionized distilled, to conform to ASTM Specification D 1193-74, Type 3. If high concentrations of organic matter are not expected to be present, the analyst may delete the potassium permanganate test for oxidizable organic matter.

4.1.4 Nitric Acid, 0.1 N. Dilute 0.5 ml of concentrated HNO_3 to 1 liter with deionized distilled water. (It may be desirable to run blanks before field use to eliminate a high blank on test samples.)

4.2 Pretest Preparation: 6 N HNO_3 is needed. Dilute 397 ml of concentrated HNO_3 to 1 liter with deionized distilled water.

4.3 Sample Recovery: 0.1 N HNO_3 [same as 4.1.4 above] is needed for sample recovery.

4.4 Analysis: The following reagents are needed for analysis (use ACS reagent grade chemicals or equivalent, unless otherwise specified):

4.4.1 Water. Same as 4.1.3 above.

4.4.2 Nitric Acid, Concentrated.

4.4.3 Nitric Acid, 50 percent (V/V). Dilute 500 ml of concentrated HNO_3 to 1 liter with deionized distilled water.

4.4.4 Stock Lead Standard Solution, 1000 $\mu\text{g Pb/ml}$. Dissolve 0.1598 g of lead nitrate [$\text{Pb}(\text{NO}_3)_2$] in about 60 ml of deionized distilled water, add 2 ml concentrated HNO_3 , and dilute to 100 ml with deionized distilled water.

4.4.5 Working Lead Standards. Pipet 0.0, 1.0, 2.0, 3.0, 4.0, and 5.0 ml of the stock lead standard solution (4.4.4) into 250-ml volumetric flasks. Add 5 ml of concentrated HNO_3 to each flask and dilute to volume with deionized distilled water. These working standards contain 0.0, 4.0, 8.0, 12.0, 16.0, and 20.0 $\mu\text{g Pb/ml}$, respectively. Prepare, as needed, additional standards at other concentrations in a similar manner.

4.4.6 Air. Suitable quality for atomic absorption analysis.

4.4.7 Acetylene. Suitable quality for atomic absorption analysis.

4.4.8 Hydrogen Peroxide, 3 percent (V/V). Dilute 10 ml of 30 percent H_2O_2 to 100 ml with deionized distilled water.

5. Procedure:

5.1 Sampling: The complexity of this method is such that, in order to obtain reliable results, testers should be trained and experienced with the test procedures.

5.1.1 Pretest Preparation: Follow the same general procedure given in Method 5, Section 4.1.1, except the filter need not be weighed.

5.1.2 Preliminary Determinations: Follow the same general procedure given in Method 5, Section 4.1.2.

5.1.3 Preparation of Collection Train: Follow the same general procedure given in Method 5, Section 4.1.3, except place 100 ml of 0.1 HNO_3 in each of the first two impingers, leave the third impinger empty, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the fourth impinger. Set up the train as shown in Figure 13-1.

5.1.4 Leak-Check Procedures: Follow the general leak-check procedures given in Method 5, Sections 4.1.4.1 (Pretest Leak-Check), 4.1.4.2 (Leak-Checks During the Sample Run), and 4.1.4.3 (Post-Test Leak-Check).

5.1.5 Sampling Train Operation: Follow the same general procedure given in Method 5, Section 4.1.5. For each run, record the data required on a data sheet such as the one shown in EPA Method 5, Figure 5-2.

5.1.6 Calculation of Percentokinetic: Same as Method 5, Section 4.1.6.

5.2 Sample Recovery: Begin proper cleanup procedure as soon as the probe is removed from the stack at the end of the sampling period.

Allow the probe to cool. When it can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it. Do not cap off the probe tip tightly while the sampling train

is cooling down as this would create a vacuum in the filter holder, thus drawing liquid from the impingers into the filter.

Before moving the sampling train to the cleanup site, remove the probe from the sampling train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Wipe off the silicone grease from the glassware inlet where the probe was fastened and cap the inlet. Remove the umbilical cord from the last impinger and cap the impinger. The tester may use ground-glass stoppers, plastic caps, or serum caps to close these openings.

Transfer the probe and filter-impinger assembly to a cleanup area, which is clean and protected from the wind so that the chances of contaminating or losing the sample are minimized.

Inspect the train prior to and during disassembly and note any abnormal conditions. Treat the samples as follows:

5.2.1 Container No. 1 (Filter): Carefully remove the filter from the filter holder and place it in its identified petri dish container. If it is necessary to fold the filter, do so such that the sample-exposed side is inside the fold. Carefully transfer to the petri dish any visible sample matter and/or filter fibers that adhere to the filter holder gasket by using a dry Nylon bristle brush and/or a sharp-edged blade. Seal the container.

5.2.2 Container No. 2 (Probe): Taking care that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover sample matter or any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components with 0.1 N HNO_3 , and placing the wash into a glass sample storage container. Measure and record (to the nearest 2-ml) the total amount of 0.1 N HNO_3 used for each rinse. Perform the 0.1 N HNO_3 rinses as follows:

Carefully remove the probe nozzle and rinse the inside surfaces with 0.1 N HNO_3 from a wash bottle while brushing with a stainless steel, Nylon-bristle brush. Brush until the 0.1 N HNO_3 rinse shows no visible particles, then make a final rinse of the inside surface.

Brush and rinse with 0.1 N HNO_3 the inside parts of the Swagelok fitting in a similar way until no visible particles remain.

Rinse the probe liner with 0.1 N HNO_3 . While rotating the probe so that all inside surfaces will be rinsed with 0.1 N HNO_3 , tilt the probe and squirt 0.1 N HNO_3 into its upper end. Let the 0.1 N HNO_3 drain from the lower end into the sample container. The tester may use a glass funnel to aid in transferring liquid wastes to the container. Follow the rinse with a probe brush. Hold the probe in an inclined position, squirt 0.1 N HNO_3 into the upper end of the probe as the probe brush is being pushed with a sawing action through the probe; hold the sample container underneath the lower end of the probe and catch any 0.1 N HNO_3 and sample matter that is brushed from the probe. Run the brush through the probe three times or more until no visible sample matter is carried out with the 0.1 N HNO_3 , and make a final visual inspection with

*Mention of trade names or specific products does not constitute endorsement by the U.S. Environmental Protection Agency.

stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times, since metal probes have small crevices in which sample matter can be entrapped. Rinse the brush with 0.1 N HNO₃ and quantitatively collect these washings in the sample container. After the brushing make a final rinse of the probe as described above.

It is recommended that two people clean the probe to minimize loss of sample. Between sampling runs, keep brushes clean and protected from contamination.

After insuring that all joints are wiped clean of silicone grease, brush and rinse with 0.1 N HNO₃ the inside of the front half of the filter holder. Brush and rinse each surface three times or more, if needed, to remove visible sample matter. Make a final rinse of the brush and filter holder. After all 0.1 N HNO₃ washings and sample matter are collected in the sample container, tighten the lid on the sample container so that the fluid will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether leakage occurs during transport. Label the container to clearly identify its contents.

5.2.3 Container No. 3 (Silica Gel). Check the color of the indicating silica gel to determine if it has been completely spent and make a notation of its condition. Transfer the silica gel from the fourth impinger to the original container and seal. The tester may use a funnel to pour the silica gel and a rubber policeman to remove the silica gel from the impinger. It is not necessary to remove the small amount of particles that may adhere to the walls and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, the tester may follow procedure for Container No. 3 under Section 5.4 (Analysis).

5.2.4 Container No. 4 (Impingers). Due to the large quantity of liquid involved, the tester may place the impinger solutions in several containers. Clean each of the first three impingers and connecting glassware in the following manner:

1. Wipe the impinger ball joints free of silicone grease and cap the joints.
2. Rotate and agitate each impinger, so that the impinger contents might serve as a rinse solution.
3. Transfer the contents of the impingers to a 500-ml graduated cylinder. Remove the outlet ball joint cap and drain the contents through this opening. Do not separate the impinger parts (inner and outer tubes) while transferring their contents to the cylinder. Measure the liquid volume to within ± 2 ml. Alternatively, determine the weight of the liquid to within ± 0.5 g. Record in the log the volume or weight of the liquid present, along with a notation of any color or film observed in the impinger catch. The liquid volume or weight is needed, along with the silica gel data, to calculate the stack gas moisture content (see Method 5, Figure 5-3).

4. Transfer the contents to Container No. 4.

5. Note: In steps 5 and 6 below, measure and record the total amount of 0.1 N HNO₃ used for rinsing. Pour approximately 30 ml of 0.1 N HNO₃ into each of the first three

impingers and agitate the impingers. Drain the 0.1 N HNO₃ through the outlet arm of each impinger into Container No. 4. Repeat this operation a second time; inspect the impingers for any abnormal conditions.

6. Wipe the ball joints of the glassware connecting the impingers free of silicone grease and rinse each piece of glassware twice with 0.1 N HNO₃; transfer this rinse into Container No. 4. (Do not rinse or brush the glass-fritted filter support.) Mark the height of the fluid level to determine whether leakage occurs during transport. Label the container to clearly identify its contents.

5.2.5 Blanks. Save 200 ml of the 0.1 N HNO₃ used for sampling and cleanup as a blank. Take the solution directly from the bottle being used and place into a glass sample container labeled "0.1 N HNO₃ blank."

5.3 Sample Preparation.

5.3.1 Container No. 1 (Filter). Cut the filter into strips and transfer the strips and all loose particulate matter into a 125-ml Erlenmeyer flask. Rinse the petri dish with 10 ml of 50 percent HNO₃ to insure a quantitative transfer and add to the flask. (Note: If the total volume required in Section 5.3.3 is expected to exceed 80 ml, use a 250-ml Erlenmeyer flask in place of the 125-ml flask.)

5.3.2 Containers No. 2 and No. 4 (Probe and Impingers). (Check the liquid level in Containers No. 2 and/or No. 4 and confirm as to whether or not leakage occurred during transport; note observation on the analysis sheet. If a noticeable amount of leakage had occurred, either void the sample or take steps, subject to the approval of the Administrator, to adjust the final results.) Combine the contents of Containers No. 2 and No. 4 and take to dryness on a hot plate.

5.3.3 Sample Extraction for lead. Based on the approximate stack gas particulate concentration and the total volume of stack gas sampled, estimate the total weight of particulate sample collected. Then transfer the residue from Containers No. 2 and No. 4 to the 125-ml Erlenmeyer flask that contains the filter using rubber policeman and 10 ml of 50 percent HNO₃ for every 100 mg of sample collected in the train or a minimum of 30 ml of 50 percent HNO₃, whichever is larger.

Place the Erlenmeyer flask on a hot plate and heat with periodic stirring for 30 min at a temperature just below boiling. If the sample volume falls below 15 ml, add more 50 percent HNO₃. Add 10 ml of 3 percent H₂O₂ and continue heating for 10 min. Add 50 ml of hot (80°C) deionized distilled water and heat for 20 min. Remove the flask from the hot plate and allow to cool. Filter the sample through a Millipore membrane filter or equivalent and transfer the filtrate to a 250-ml volumetric flask. Dilute to volume with deionized distilled water.

5.3.4 Filter Blank. Determine a filter blank using two filters from each lot of filters used in the sampling train. Cut each filter into strips and place each filter in a separate 125-ml Erlenmeyer flask. Add 15 ml of 50 percent HNO₃ and treat as described in Section 5.3.3 using 10 ml of 3 percent H₂O₂ and 50 ml of hot deionized distilled water. Filter and dilute to a total volume of 100 ml using deionized distilled water.

5.3.5 0.1 N HNO₃ Blank. Take the entire 200 ml of 0.1 N HNO₃ to dryness on a steam

bath, add 15 ml of 50 percent HNO₃ and treat as described in Section 5.3.3 using 10 ml of 3 percent H₂O₂ and 50 ml of hot deionized distilled water. Dilute to a total volume of 100 ml using deionized distilled water.

5.4 Analysis.

5.4.1 Lead Determination. Calibrate the spectrophotometer as described in Section 8.2 and determine the absorbance for each source sample, the filter blank, and 0.1 N HNO₃ blank. Analyze each sample three times in this manner. Make appropriate dilutions, as required, to bring all sample Pb concentrations into the linear absorbance range of the spectrophotometer.

If the Pb concentration of a sample is at the low end of the calibration curve and high accuracy is required, the sample can be taken to dryness on a hot plate and the residue dissolved in the appropriate volume of water to bring it into the optimum range of the calibration curve.

5.4.2 Mandatory Check for Matrix Effects on the Lead Results. The analysis for Pb by atomic absorption is sensitive to the chemical composition and to the physical properties (viscosity, pH) of the sample (matrix effects). Since the Pb procedure described here will be applied to many different sources, many sample matrices will be encountered. Thus, check (mandatory) at least one sample from each source using the Method of Additions to ascertain that the chemical composition and physical properties of the sample did not cause erroneous analytical results.

Three acceptable "Method of Additions" procedures are described in the General Procedure Section of the Perkin Elmer Corporation Manual (see Citation 9.1). If the results of the Method of Additions procedure on the source sample do not agree within 5 percent of the value obtained by the conventional atomic absorption analysis, then the tester must reanalyze all samples from the source using the Method of Additions procedure.

5.4.3 Container No. 3 (Silica Gel). The tester may conduct this step in the field. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g; record this weight.

6. Calibration.

Maintain a laboratory log of all calibrations.

6.1 Sampling Train Calibration. Calibrate the sampling train components according to the indicated sections of Method 5: Probe Nozzle (Section 5.1); Pitot Tube (Section 5.2); Metering System (Section 5.3); Probe Heater (Section 5.4); Temperature Gauges (Section 5.5); Leak-Check of the Metering System (Section 5.6); and Barometer (Section 5.7).

6.2 Spectrophotometer. Measure the absorbance of the standard solutions using the instrument settings recommended by the spectrophotometer manufacturer. Repeat until good agreement (± 3 percent) is obtained between two consecutive readings. Plot the absorbance (y-axis) versus concentration in $\mu\text{g Pb/ml}$ (x-axis). Draw or compute a straight line through the linear portion of the curve. Do not force the calibration curve through zero, but if the curve does not pass through the origin or at least be closer to the origin than ± 0.003

For absorbance units, check for incorrectly prepared standards and for curvature in the calibration curve.

To determine stability of the calibration curve, run a blank and a standard after every five samples and recalibrate, as necessary.

7. Calculations.

7.1 Dry Gas Volume. Using the data from this test, calculate V_{meters} , the total volume of dry gas metered corrected to standard conditions (20°C and 760 mm Hg), by using Equation 5-1 of Method 5. If necessary, adjust V_{meters} for leakages as outlined in Section 6.3 of Method 5. See the field data sheet for the average dry gas meter temperature and average orifice pressure drop.

7.2 Volume of Water Vapor and Moisture Content. Using data obtained in this test and Equations 5-2 and 5-3 of Method 5, calculate the volume of water vapor (V_{water}) and the moisture content B_w of the stack gas.

7.3 Total Lead in Source Sample. For each source sample correct the average absorbance for the contribution of the filter blank and the 0.1 N HNO₃ blank. Use the calibration curve and this corrected absorbance to determine the µg Pb concentration in the sample aspirated into the spectrophotometer. Calculate the total Pb content C_m (in µg) in the original source sample; correct for all the dilutions that were made to bring the Pb concentration of the sample into the linear range of the spectrophotometer.

7.4 Lead Concentration. Calculate the stack gas Pb concentration C_n in mg/dscm as follows:

$$C_n = K \frac{C_m}{V_{\text{meters}}}$$

Where:

$K = 0.001$ mg/µg for metric units,
 $= 2.205$ lb/µg for English units.

7.5 Isokinetic Variation and Acceptable Results. Same as Method 5, Sections 6.11 and 6.12, respectively. To calculate v_a , the average stack gas velocity, use Equation 2-9 of Method 2 and the data from this field test.

8. Alternative Test Methods for Inorganic Lead.

8.1 Simultaneous Determination of Particulate and Lead Emissions. The tester may use Method 5 to simultaneously determine Pb provided that (1) he uses acetone to remove particulate from the probe and inside of the filter holder as specified by Method 5, (2) he uses 0.1 N HNO₃ in the impingers, (3) he uses a glass fiber filter with a low Pb background, and (4) he treats and analyzes the entire train contents, including the impingers, for Pb as described in Section 5 of this method.

8.2 Filter Location. The tester may use a filter between the third and fourth impinger provided that he includes the filter in the analysis for Pb.

8.3 In-stack Filter. The tester may use an in-stack filter provided that (1) he uses a

glass-lined probe and at least two impingers, each containing 100 ml of 0.1 N HNO₃, after the in-stack filter and (2) he recovers and analyzes the probe and impinger contents for Pb. Recover sample from the nozzle with acetone if a particulate analysis is to be made.

9. Bibliography

9.1 Perkin Elmer Corporation. Analytical Methods for Atomic Absorption Spectrophotometry. Norwalk, Connecticut, September 1976.

9.2 American Society for Testing and Materials. Annual Book of ASTM Standards, Part 31: Water, Atmospheric Analysis. Philadelphia, Pa. 1974. p. 40-42.

9.3 Klein, R. and C. Hoch. Standard Additions—Uses and Limitations in Spectrophotometric Analysis. *Amer. Lab.* 9:21-27, 1977.

9.4 Mitchell, W.J. and M.R. Midgett. Determining Inorganic and Alkyl Lead Emissions from Stationary Sources. U.S. Environmental Protection Agency, Emission Monitoring and Support Laboratory, Research Triangle Park, N.C. (Presented at National APCA Meeting, Houston, June 20, 1978).

9.5 Same as Method 5, Citations 2 to 5 and 7 of Section 7.

(Secs. 111, 114, and 301(a) of the Clean Air Act as amended (42 U.S.C. 7411, 7414, and 7601(a)))

(EPA Doc. #2-10441 Filed 4-15-82; 4:43 pm)

ENVIRONMENTAL PROTECTION AGENCY

40 CFR Part 60

[AD-FRL-2620-3] *ASPS*

Standards of Performance for New Stationary Sources; Reference Methods; Revision to Method 12 for Inorganic Lead To Add a Method of Additions Procedure

AGENCY: Environmental Protection Agency (EPA).

ACTION: Final rule.

SUMMARY: The purpose of this action is to promulgate a revision to Method 12 for inorganic lead of Appendix A of 40 CFR Part 60 to include a method of additions procedure, which deals with the resolution of any possible interferences in the lead analysis. This revision is necessary because it has been determined that the method of additions procedures previously cited by Method 12 may not be readily available to the analyst, and were not suitable for incorporation by reference. This revision was proposed in the Federal Register on December 12, 1983 (48 FR 55395). No changes in the revision have been made since proposal, as no comment letters were received.

EFFECTIVE DATE: August 24, 1984.

Under section 307(b)(1) of the Clean Air Act, judicial review of this revision is available *only* by the filing of a petition for review in the U.S. Court of Appeals for the District of Columbia Circuit within 60 days of today's publication of this rule. Under section 307(b)(2) of the Clean Air Act, the requirements that are the subject of today's notice may not be challenged later in civil or criminal proceedings brought by EPA to enforce these requirements.

ADDRESSES: *Docket.* A docket, number A-83-38, containing materials considered by EPA in development of the promulgated rulemaking, is available for public inspection between 8:00 a.m. and 4:00 p.m., Monday through Friday, at EPA's Central Docket Section (LE-131), West Tower Lobby, Gallery 1, 401 M Street, SW., Washington, D.C. 20460. A reasonable fee may be charged for copying.

FOR FURTHER INFORMATION CONTACT: William Grimley or Roger Shigehara, Emission Measurement Branch, Emission Standards and Engineering Division (MD-19), U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711, telephone (919) 541-2237.

SUPPLEMENTARY INFORMATION: This rulemaking does not impose any additional emission measurement requirements on facilities affected by this rulemaking. Rather, this rulemaking adds a supplementary analytical procedure to a test method that would apply irrespective of this rulemaking. This addition is necessary because the supplementary analytical procedure, which was previously cited by the method, is not suitable for incorporation by reference.

Public Participation

The revision was proposed and published in the Federal Register on December 12, 1983 (48 FR 55395). The opportunity to request a public hearing was presented to provide interested persons the opportunity for oral presentation of data, views, or arguments concerning the proposed revision, but no person desired to make an oral presentation. The public comment period was from December 12, 1983, to February 27, 1984. No comment letters were received.

Docket

The docket is an organized and complete file of the information considered by EPA in the development of this rulemaking. The docket is a dynamic file, since material is added throughout the rulemaking development. The docketing system is intended to allow members of the public and industries involved to identify readily and locate documents so that they can intelligently and effectively participate in the rulemaking process. Along with the statement of basis and purpose of the proposed and promulgated rule and EPA responses to significant comments, the contents of the docket will serve as the record in case of judicial review (Section 307(d)(7)(A)).

Miscellaneous

Under Executive Order 12291, EPA must judge whether a regulation is "major" and therefore subject to the requirement of a regulatory impact analysis. This regulation is not major because it will not have an annual effect on the economy of \$100 million or more; it will not result in a major increase in costs or prices; and there will be no significant adverse effects on competition, employment, investment, productivity innovation, or on the ability of U.S.-based enterprises to compete with foreign-based enterprises in domestic or export markets. This regulation was submitted to OMB for review under E.O. 12291.

Pursuant to the provisions of 5 U.S.C. 605(u), I hereby certify that the attached

rule will not have a significant economic impact on small entities because no additional costs will be incurred. This rule does not contain any information collection requirements subject to OMB review under the Paperwork Reduction Act of 1980 U.S.C. 3501 *et seq.*

This rulemaking is issued under the authority of sections 111, 114, and 301(a) of the Clean Air Act, as amended (42 U.S.C. 7411, 7414, and 7601(a)).

List of Subjects in 40 CFR Part 60

Air pollution control, Aluminum, Ammonium sulfate plants, Asphalt, Cement industry, Coal Copper, Electric power plants, Glass and glass products, Grains, Intergovernmental relations, Iron, Lead, Metals, Metallic Minerals, Motor vehicles, Nitric acid plants, Paper and paper products industry, Petroleum, Phosphate, Sewage disposal, Steel Sulfuric acid plants, Waste treatment and disposal, Zinc, Tires, Incorporation by Reference, Can surface coating, Sulfuric acid plants, Industrial organic chemicals, Organic solvent cleaners, Fossil fuel-fired steam generators, Fiberglass Insulation, Synthetic Fibers, Lime.

Dated: August 2, 1984.

Alvin L. Alm,

Acting Administrator.

PART 60—[AMENDED]

40 CFR Part 60, Appendix A, Method 12, is amended as follows:

1. By revising Section 5.4.2 to read as follows:

§ 5.4.2 Check for Matrix Effects on the Lead Results.

Since the analysis for Pb by atomic absorption is sensitive to the chemical composition and to the physical properties (viscosity, pH) of the sample (matrix effects), the analyst shall check at least one sample from each source using the method of additions as follows:

Add or spike an equal volume of standard solution to an aliquot of the sample solution, then measure the absorbance of the resulting solution and the absorbance of an aliquot of unspiked sample.

Next, calculate the Pb concentration C_s in $\mu\text{g}/\text{ml}$ of the sample solution by using the following equation:

$$C_s = C_a \frac{A_s}{A_s - A_u} \quad \text{Eq. 12-1}$$

Where:

C_s = Pb concentration of the standard solution, $\mu\text{g}/\text{ml}$.

rate tables, which are proposed amendments to the International Mail Manual (incorporated by reference in the Code of Federal Regulations, 39 CFR 10.1), and which are to become effective on the date service begins. No comments were received.

Accordingly, the Postal Service states that it intends to begin Express Mail International Service with Norway on October 23, 1984 at the rates indicated in the table below.

List of Subjects in 39 CFR Part 10

Postal service, Foreign relations.

NORWAY.—EXPRESS MAIL INTERNATIONAL SERVICE

Custom designed service ¹		On demand service ²	
Up to and including		Up to and including	
Pounds	Rate	Pounds	Rate
1	\$28.00	1	\$28.00
2	31.70	2	23.70
3	35.40	3	27.40
4	38.10	4	31.10
5	42.80	5	34.80
6	46.50	6	38.50
7	50.20	7	42.20
8	53.90	8	45.90
9	57.60	9	49.60
10	61.30	10	53.30
11	65.00	11	57.00
12	68.70	12	60.70
13	72.40	13	64.40
14	76.10	14	68.10
15	79.80	15	71.80
16	83.50	16	75.50
17	87.20	17	79.20
18	90.90	18	82.90
19	94.60	19	86.60
20	98.30	20	90.30
21	102.00	21	94.00
22	105.70	22	97.70
23	109.40	23	101.40
24	113.10	24	105.10
25	116.80	25	108.80
26	120.50	26	112.50
27	124.20	27	116.20
28	127.90	28	119.90
29	131.60	29	123.60
30	135.30	30	127.30
31	139.00	31	131.00
32	142.70	32	134.70
33	146.40	33	138.40
34	150.10	34	142.10
35	153.80	35	145.80
36	157.50	36	149.50
37	161.20	37	153.20
38	164.90	38	156.90
39	168.60	39	160.60
40	172.30	40	164.30
41	176.00	41	168.00
42	179.70	42	171.70
43	183.40	43	175.40
44	187.10	44	179.10

¹ Rates in this table are applicable to each piece of International Custom Designed Express Mail shipped under a Service Agreement providing for tender by the customer at a designated Post Office.

² Pickup is available under a Service Agreement for an added charge of \$5.00 for each pickup stop, regardless of the number of pieces picked up. Domestic and International Express Mail picked up together under the same Service Agreement incurs only one pickup charge.

A transmittal letter making these changes in the pages of the International Mail Manual will be published in the Federal Register as provided in 39 CFR 10.3 and will be transmitted to subscribers automatically.

(39 U.S.C. 401, 404, 407)
W. Allen Sanders,
Associate General Counsel, Office of General
Law and Administration.
[FR Doc. 84-25247 Filed 9-21-84; 9:45 am]
BILLING CODE 7710-12-M

ENVIRONMENTAL PROTECTION AGENCY

40 CFR Part 60

[AD-FRL-2620-3]

Standards of Performance for New Stationary Sources; Reference Methods; Revision To Method 12 for Inorganic Lead To Add a Method of Additions Procedure

Correction

In FR Doc. 84-21131 beginning on page 33842 in the issue of Friday, August 24, 1984, make the following correction: In column three, the equation at the bottom of the page should read:

$$C_s = C_a \frac{A_s}{A_t - A_s}$$

BILLING CODE 7050-01-M

DEPARTMENT OF TRANSPORTATION

Coast Guard

46 CFR Part 170

[CGD 79-023]

Subdivision and Stability Regulations

AGENCY: Coast Guard, DOT.

ACTION: Final rule; correction.

SUMMARY: This document corrects three incorrect definitions in the final rule issued November 4, 1983.

FOR FURTHER INFORMATION CONTACT: Lt. Albert W. Horsmon Jr., Commandant (G-MTH-5/13), Room 1308, U.S. Coast Guard Headquarters, Washington, D.C. 20593, (202) 426-2187.

Discussion of Correction

§ 170.170 [Amended]

In the Federal Register of November 4, 1983, page 51014, some definitions for the coefficient P in the inequality defining GM in § 170.170(a) are incorrect due to typographical errors. The second, third, and fourth definitions for P are corrected to read:

$P = .055 + (L/1309)^2$ metric tons/m³ . . . for ocean service, Great Lakes winter service, or service on exposed waters.

$P = .0033 + (L/14,200)^2$ tons/ft³ . . . for Great Lakes summer service or service on partially protected waters.
 $P = .036 + (L/1309)^2$ metric tons/m³ . . . for Great lakes summer service or service on partially protected waters.

Dated: September 18, 1984.

Clyde T. Lusk, Jr.,
Rear Admiral, U.S. Coast Guard, Chief, Office of Merchant Marine Safety.

[FR Doc. 84-25212 Filed 9-21-84; 9:45 am]

BILLING CODE 4910-14-M

FEDERAL COMMUNICATIONS COMMISSION

47 CFR Part 1

Amendment of Part 1 of the Rules Concerning Practice and Procedure in the Private Radio Services

AGENCY: Federal Communications Commission.

ACTION: Final rule; correction.

SUMMARY: This document corrects an error in the Appendix to an amendment of the rules of practice and procedure in the Private Radio Services.

FOR FURTHER INFORMATION CONTACT: Robert DeYoung, Private Radio Bureau, (202) 632-7175

Mary Beth Hess, Private Radio Bureau, (202) 634-2443

SUPPLEMENTARY INFORMATION

Erratum

Released: September 17, 1984.

In the matter of amendment of Part 1 of the Rules concerning Practice and Procedure in the Private Radio Services.

On July 24, 1984, the Commission released an Order (FCC 84-323) (August 2, 1984, 49 FR 30943) in the above-captioned proceeding. Section 1.925(f), (g) and (h) were printed incorrectly in the Appendix. Those paragraphs should read:

§ 1.925 Application for special temporary authorization, temporary permit or temporary operating authority.

(f) An applicant for a Ship Radio station license may operate the radio station pending issuance of the ship station authorization by the Commission for a period of 90 days, under a temporary operating authority, evidenced by a properly executed certification made on FCC Form 506-A.

(g) An applicant for a Business Radio station license utilizing an already authorized facility may operate the radio station for a period of 180 days, under a temporary permit, evidenced by